

08/48664-22



PATENT
IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

re Application of

Klaus W. Hartig, et al.

Serial No.: (Cont. Appln. of
S.N. 08/102,281 filed 8/5/93)

Filed: (concurrently herewith)

For: HEAT TREATABLE, DURABLE,
IR-REFLECTING SPUTTER-
COATED GLASSES AND METHOD
OF MAKING SAME

Examiner:

Group Art Unit: 1508

Atty. Dkt. No.: 2372.853

PETITION TO EXPUNGE CONFIDENTIAL INFORMATION
(37 C.F.R. §§1.97, 1.56 AND M.P.E.P 724.02, .05)

Honorable Commissioner of
Patents and Trademarks
Washington, D.C. 20231

Sir:

Now come the above-identified Applicants and petition the
Honorable Commissioner of Patents pursuant to M.P.E.P. 724.02 to
expunge certain proprietary information found in the sealed
envelope attached to this Petition, from the record after
consideration thereof by the Examiner during his examination of
this application.

Pursuant to the aforesaid section 724.02 M.P.E.P., the
envelope in which the confidential information is found has been
sealed and has been clearly labeled as such by being marked
"CONFIDENTIAL MATERIAL NOT OPEN TO THE PUBLIC", "TO BE OPENED
ONLY BY EXAMINER OR OTHER AUTHORIZED PATENT AND TRADEMARK OFFICE
EMPLOYEE".

The information contained therein consists of 2 documents, the first, a twenty-two page document and the second, a twenty-three page document, both reflecting certain alleged confidential information of a third party, Airco Corporation, related to its commercially available products known as Super-E III and Super-E IV. Attached to the IDS submitted simultaneously herewith there is provided a redacted version of each of these two documents. The only information redacted out is any alleged proprietary information. Left in is what is believed to be only non-proprietary and non-confidential information. These redacted versions, freely available to the public upon issuance of a patent herefrom, are submitted for the Examiner's comparative consideration.

It is respectfully submitted that, given the redacted versions, this Petition may be granted because none of the proprietary information (as perhaps opposed to the non-proprietary information) will be found by the Examiner to be relevant or material to the Examiner's deliberations. We, of course, leave to the Examiner, the ultimate determination as to this issue.

Further pursuant to the provisions of M.P.E.P. 724.05, the six criteria set forth therein for permitting such a petition, are herein fulfilled as follows:

1. A clear identification of the information to be expunged without disclosure of the details thereof has been



presented above and is found in the clearly marked envelope attached hereto.

2. A clear statement of the information to be expunged and subject to the Protective Order is the full text of the documents found in the sealed envelope as compared to the freely available, redacted form of each found in the IDS. On information and belief, the comparative information allegedly proprietary to Airco, has not been otherwise made available to the public.

3. Since this application has not yet been examined, it is not possible to identify the application paper which held the information to be important to decide to the issue of patentability. We respectfully request the Examiner to consider this Petition and the confidential information during his deliberations.

4. Petitioners hereby agree to retain the information in the enclosed sealed envelope for the length of time of any patent with regard to which such information is submitted upon grant of this Petition and the return of the sealed envelope and its contents to the undersigned.


5. The Petition to Expunge, along with the allegedly confidential information in the sealed envelope, is being submitted herewith by the Applicants and their assignee, Guardian Industries Corporation. The information is that of a third party Airco Corporation.

6. Pursuant to 37 C.F.R. §1.17(h), the requisite fee in the amount of \$130.00 for a petition under 37 C.F.R. §1.182 is

submitted herewith. Should any additional monies be necessary to cover this fee, the United States Patent and Trademark Office is authorized herewith to charge that additional amount to Applicants' account no. 13-5132.

Respectfully submitted,

Dated: June 7, 1995.


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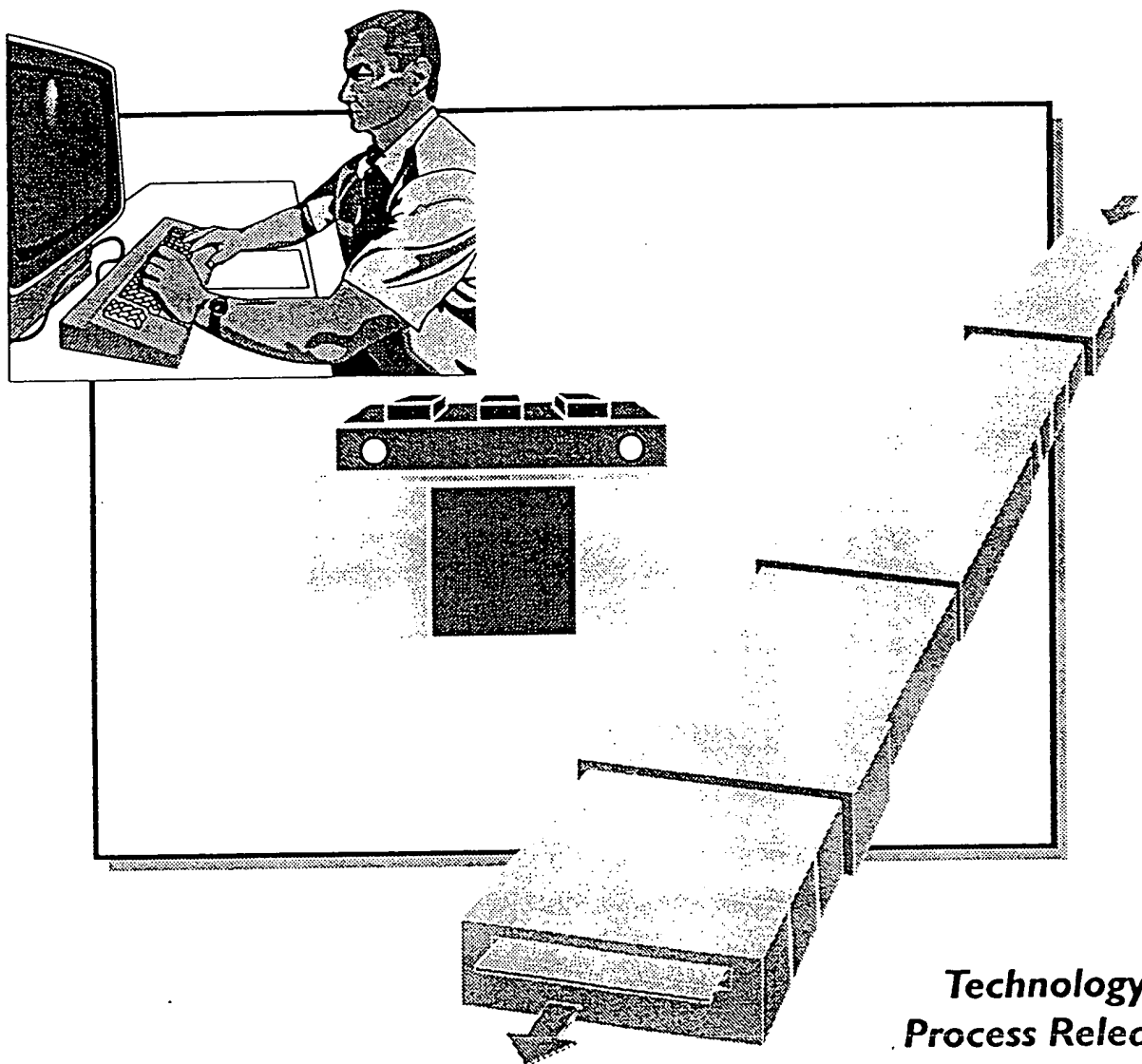
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Super-E™ IV



Technology
Process Release

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I. Introduction

A new, durable low-emissivity film has been developed by Airco Coating Technology. The film has higher durability than the Super-E™ II coating, and it has higher visible transmittance than Super-E™ III, (see Table 1). This has been accomplished by reducing the as-deposited stress in the Si_3N_4 layer (made possible by the C-MAG® sputtering cathode), by process improvements in the barrier technology, and by the use of TiO_2 as the first layer. The emissivity of this Low-E stack has also been lowered to 0.10 or less as calculated for normal emissivity based on specular reflectance measurements between 5-15 microns.

The following coating release details the coating stack design, optical specification, durability testing, and process cycle time estimates.

II. Coating Design

The layer design for Super-E IV coating is as follows:

Glass	TiO_2	NiCrN_x	Ag	NiCrN_x	Si_3N_4
	~300 Å	~8 Å	~105 Å	~8 Å	~425 Å

The thickness of the NiCrN_x barrier layers is approximate and was estimated using optical modeling techniques.

III. Optical Performance

Table 2 contains a summary of the optical properties for eight samples of Super-E IV made in Airco's flat glass development coater, G-6, on 3 mm clear glass substrates and includes a specification for Super-E IV on 3 mm clear glass. The emissivity was measured on a PE-1420 IR spectrophotometer, and was based on specular reflectance measurements between 5-15 microns. A graph of reflection and transmission for a typical sample in the visible region is shown in Figure 1. Thin film modeling techniques were used to calculate optical performance data for Super-E IV on 4 mm and 6 mm glass thicknesses, these results are shown in Table 3. Illuminant C (10 degree observer angle) was used for both measurement and calculation of optical performance data. Table 4 contains a listing of the optical properties at selected wavelengths for the 3, 4, and 6 mm glass used for the calculations.

Super-E IV IGU thermal data has been calculated with a program developed by the University of California, Lawrence Berkeley Laboratory titled, Window 4.0. Tables 5, 6, and 7 contain the data calculated for 3, 4, and 6 mm glass respectively, along with assumptions made for calculations of the IGU characteristics. Data are presented in both English and Metric units for each glass thickness.

IV. Durability Testing

Durability testing of the coatings produced in the G-6 development system consisted of an abrasion test and several corrosion tests. Appendix 1 details the tests administered and the method of evaluation for each test. The tests included the following: humidity exposure, salt fog exposure, UV exposure, salt dot, ammonium hydroxide immersion, sodium hydroxide immersion, hydrochloric acid immersion, sulfuric acid immersion, nitric acid immersion and Taber abrasion. The strengths of solutions, temperatures, times etc. are contained in the test descriptions of Appendix 1. Summarized data from durability testing of the eight samples of Super-E IV are presented in Table 8, detailed test data are available on request. Airco cannot make claims to performance of Super-E IV coatings in other types of durability tests. The test data demonstrate the comparable durability of Super-E III and Super-E IV, and the greatly improved durability over Super-E II and other standard low-emissivity coatings.

V. Process Description

Table 9 shows a typical deposition process used in the development work performed in the G-6 system. The TiO_2 layer was deposited utilizing planar cathodes, however a C-MAG rotatable cathode would be recommended for higher production deposition rates, in an O_2 / Ar gas mixture. The NiCrN_x barrier layers were deposited in a N_2 / Ar gas mixture, and a 3 inch wide slit was used to limit the deposition rate of the NiCrN_x . The thinness of the barrier layer requires precise control of the deposition rate, and the use of a deposition mask is a technique available to improve thickness control. The Ag metal layer was also sputtered in a N_2 / Ar gas mixture, but no slit was required for control of the silver thickness. The Si_3N_4 layer was deposited in a N_2 / Ar / He gas mixture at a higher process pressure of 4 milli-torr. The magnet bars of the C-MAG cathodes were rotated to a position of 45 degrees, pointing inward. Figure 2 shows the layout of the rotated magnet bar C-MAG cathode in the G-6 development system. End shields were modified for use with the rotated magnet configuration. Reduction of the as-deposited intrinsic stress in the silicon nitride and optimization of the barrier thickness was accomplished with these modifications, when compared to the Si_3N_4 process of Super-E III. All cathodes require floating anodes.

VI. Cycle Time Calculations

Estimated cycle times for Super-E IV were calculated for a variety of system configurations. Table 10 contains the coating design, deposition rates, maximum power densities, and maximum powers for both the C-MAG 6000 and C-MAG 7200 series cathodes. The Dynamic Deposition Rate (DDR) was measured in the G-6 development system and is a function of cathode power, line speed, thickness deposited, number of targets, and racetrack length. Table 11 shows maximum operating power conditions for HRC-6000 and HRC-7200 cathodes. Tables 12 and 13 show the resulting cycle times for 3, 4 and 5 coat zone systems utilizing these cathodes. The cycle time data have been presented graphically in Figure 3 for 144-inch glass and Figure 4 for 6-meter glass.

VII. Conclusion

The Super-E IV coating design developed by Airco Coating Technology offers similar visible transmittance and greatly improved durability over our Super-E II coating; while demonstrating comparable durability to Super-E III with lower emissivity and higher transmittance. The design maintains neutrality in reflected color at all angles of incidence from 0-80 degrees.

Since the Super-E IV design requires different gas mixes and cathode configurations, it is recommended that licensees contact Airco Coating Technology before attempting to fabricate Super-E IV coatings.

Appendix 1 - Low-E (Super-E™ IV)

TEST CONDITIONS AND SCORING PROCEDURES

Revised October 1992

- | | | |
|----|-------------------------|---|
| A) | Humidity Test | 96 hours exposure in a humidity cabinet at 60°C with 95% RH |
| B) | Salt Fog Test | 72 hours in a 20% Salt Fog, at 95-98°F |
| C) | UV Exposure Test | 120 hours exposure; with cycles of 4 hours UV and 4 hours condensation. |
| D) | Salt Dot Test | A 1% (by weight) NaCl solution is applied to a filter paper square placed on the film side of the sample and the sample placed in a constant humidity environment for 24 hours. |

Tests A, B, C & D sample evaluations:

- 1) Selecting a representative area, 200X magnification photomicrographs are used to measure the defect area by the grid * technique. The defect score is calculated by the formula.

$$\text{Defect Score} = 10 - 0.5 (\% \text{ Defect Area})$$

- 2) To characterize corrosion of the metal layer, the emissivity of each sample is measured before and after the test. An emissivity score based upon change in emissivity is calculated using the following equation:

$$\text{Emissivity Score} = 10 (\text{Starting Emiss} / \text{Test Emiss})$$

Tests A, B, & C sample evaluations:

- 3) Measurement of changes in film-side reflectance and color are made. (Note: Due to heavy corrosion of the unprotected glass side in these tests, it is not possible to make glass side reflectance or transmission comparisons after the test.)

Appendix 1 - Low-E (Super-E IV)

TEST CONDITIONS AND SCORING PROCEDURES

- | | | |
|----|--|--|
| E) | NH ₄ OH Base Test | Samples are immersed in a 0.5N solution of NH ₄ OH, at room temperature, in a closed container, for 5 hours. |
| F) | NaOH Base Test | Samples are immersed in a 0.1N solution of NaOH, at room temperature, in a closed container, for 5 hours. |
| G) | HCl Acid Test | Samples are immersed in a 0.1N solution of HCl, at room temperature, in a closed container, for 5 hours. |
| H) | H ₂ SO ₄ Acid Test | Samples are immersed in a 0.1N solution of H ₂ SO ₄ , at room temperature, in a closed container, for 5 hours. |
| I) | HNO ₃ Acid Test | Samples are immersed in a 0.1N solution of HNO ₃ , at room temperature, in a closed container, for 5 hours. |

Test E, F, G, H and I samples are evaluated by:

- 1) Selecting a representative area, 200X magnification photomicrographs are used to measure the defect area by the grid * technique. The defect score is calculated by the formula.

$$\text{Defect Score} = 10 - 0.5 (\% \text{ Defect Area}).$$

- 2) Characterizing corrosion of the metal layer; the emissivity of each sample is measured. An emissivity score based upon change in emissivity is calculated using the following equation:

$$\text{Emissivity Score} = 10 (\text{Starting Emiss} / \text{Test Emiss})$$

- 3) Measuring the changes in transmission and both film- and glass-side reflectance and color.

Appendix 1 - Low-E (Super-E IV)

TEST CONDITIONS AND SCORING PROCEDURES

- J) **Taber Abrasion** Samples are subjected to a total of 50 revolutions on the Taber Abraser, using the standard 500 gram weight and CS-10F wheels.

Evaluation is based on:

- 1) Selecting a representative area on the Tabered track, a photomicrograph is taken at 50X. Using this micrograph, 4 one inch square areas are marked off and the number of scratches in each square is counted. The scratch count for the 4 areas is averaged and this average number of scratches yields a score of 0 for more than 55 scratches in a 1" square area, and 10 for none, according to the following equation:

$$\text{Taber Score} = 10 - [(\text{Average \# Scratches}) \times (0.18)]$$

- 2) The transmission in the abraded area is measured in four places and the results averaged. Using this average transmission, the changed in transmission from the unabraded area is calculated.
- 3) The haze in the abraded area is measured in four places and the results averaged. Using this average transmission, the changed in transmission from the unabraded area is calculated.

* Grid Technique

A 10 x 10 grid with a spacing of 0.5 mm is randomly placed on the micrograph. Each intersection point located within a defect is counted as 1, and a point located on a grid boundary is counted as 0.5. A score is kept for each grid placement, the process is repeated through 4 grid placements, and the number of defect points are totaled. The % defect area is then calculated using the following formula:

$$\% \text{ Defect Area} = \frac{\text{Total \# of defect points}}{\text{Total \# of points (400)}} \times 100$$

Table 1
Super-E™ Coating Comparison

All Optical Data for CIE Illuminant C at 10 Degrees Observer on 3mm coated glass
Durability Data taken from runs in Airco's flat glass Development Coater, G-6.

<u>Property</u>	<u>Super-E IV</u>	<u>Super-E III</u>	<u>Super-E II</u>
% T _{vis}	82	76	83
% R _{g vis}	6	8	10
'a'	-2.0	-2.5	-3.0
'b'	0.0	-7.5	-9.0
% R _{f vis}	4.5	4	7
'a'	-1.5	-1.0	-3.0
'b'	0.0	-6.0	-11.0
% T _{solar}	63	62	66
% R _{g solar}	16	12.5	17
% R _{f solar}	19	14	18
Emissivity	0.10	0.16	0.10
<u>Test Results</u>	(See Appendix I for test details)		
Humidity	9.5	9.4	4.5
Salt Fog	8.7	9.3	0
Taber	8.2	9.6	1.7

Table 2
Optical Performance Data
3 mm Glass Substrates

All Data were generated on a Spectrogard Color System for CIE Illuminant C at 10 Degree Observer
 G-6 Data taken from a set of 8 runs.

<u>Property</u>	<u>Measured in G-6</u>		<u>Proposed</u>
	<u>Value</u>	<u>Std. Dev</u>	<u>Specification</u>
% T _{vis}	82.0	0.53	82 ± 1
% R _{g vis}	5.9	0.72	6.0 ± 1
'a'	-2.52	0.52	-2.0 ± 1.5
'b'	0.12	0.53	0.0 ± 1.5
% R _{f vis}	4.4	0.59	4.5 ± 1
'a'	-1.48	0.69	-1.5 ± 1.5
'b'	-0.11	1.42	0.0 ± 2.5
Emissivity	0.09	.007	< 0.10

Table 3
Optical Performance Data
4mm and 6 mm Glass Substrates

All Data for CIE Illuminant "C" at 10 Degrees STD Observer

Property	3mm	4mm	6 mm
% T _{vis} in air	82 ± 1	81.5 ± 1	80 ± 1
% R _g in air	6.0 ± 1	6.0 ± 1	6.0 ± 1
'a'	-2.0 ± 1.5	-2.0 ± 1.5	-2.0 ± 1.5
'b'	0.0 ± 1.5	0.0 ± 1.5	0.0 ± 1.5
% R _f in air	4.5 ± 1	4.5 ± 1	4.5 ± 1
'a'	-1.5 ± 1.5	-1.5 ± 1.5	-1.5 ± 1.5
'b'	0.0 ± 2.5	0.0 ± 2.5	0.0 ± 2.5
% T _{solar} in air	63 ± 1	62 ± 1	59 ± 1
% R _{g solar} in air	15.5 ± 1	14.5 ± 1	12.5 ± 1
%R _{f solar} in air	19 ± 1	19 ± 1	19 ± 1
% T (IGU) (tables 5,6,&7)	74 ± 1	74 ± 1	70 ± 1
Emissivity	< 0.10	< 0.10	< 0.10
Sheet Resistance (Ohms/Square)	< 11.0	< 11.0	< 11.0

Table 4
Glass Optical Properties
Used for Models

	<u>3mm Glass</u>		<u>4mm Glass</u>		<u>6mm Glass</u>	
<u>Wavelength(nm)</u>	%R	%T	%R	%T	%R	%T
350	7.2	76.7	6.9	72.2	6.3	64.2
400	8.3	89.1	8.3	88.3	8.1	86.8
450	8.2	89.6	8.1	89.0	8.0	87.7
500	8.1	90.4	8.1	90.0	8.0	89.1
550	8.0	90.3	8.0	89.8	7.9	88.7
600	7.9	89.4	7.8	88.5	7.7	86.9
650	7.7	87.9	7.6	86.6	7.4	84.0
700	7.4	85.9	7.3	83.9	7.0	80.2
750	7.2	83.7	7.0	81.1	6.6	76.1
800	7.3	81.8	7.1	78.7	6.7	72.8
850	7.2	80.0	6.9	76.3	6.4	69.6
ILL C	8.0	89.9	7.9	89.3	7.8	88.1

Table 5
Solar / IGU Properties
3 mm Glass

	<u>English</u>	<u>Metric</u>
Shading Coefficient	0.80	0.80
Relative Heat Gain (RHG)	163	515
Winter U-Value	0.32	1.81
Summer U-Value	0.33	1.87
%T _{solar} (IGU)	54	54
%R _{solar} (IGU)	19	19

Thermal data for the IGU are based on the following assumptions:

1. The coating is glazed on the #3 surface.
2. Both lites are 3-mm-thick clear glass.
3. The gap is 0.5 inches (1.27 cm) and contains air.
4. %R_{solar} (IGU) is reflection to the outside of the building.
5. The units for RHG and U-Value are:

	<u>English</u>	<u>Metric</u>
RHG	BTU/hr-ft ²	W/m ²
U-Value	BTU/hr-ft ² -°F	W-m ² /°C

Table 6
Solar / IGU Properties
4 mm Glass

	<u>English</u>	<u>Metric</u>
Shading Coefficient	0.79	0.79
Relative Heat Gain (RHG)	161	509
Winter U-Value	0.32	1.80
Summer U-Value	0.33	1.88
%T _{solar} (IGU)	51	51
%R _{solar} (IGU)	18	18

Thermal data for the IGU are based on the following assumptions:

1. The coating is glazed on the #3 surface.
2. Both lites are 4-mm-thick clear glass.
3. The gap is 0.5 inches (1.27 cm) and contains air.
4. %R_{solar} (IGU) is reflection to the outside of the building.
5. The units for RHG and U-Value are:

	<u>English</u>	<u>Metric</u>
RHG	BTU/hr-ft ²	W/m ²
U-Value	BTU/hr-ft ² -°F	W-m ² /°C

Table 7
Solar / IGU Properties
6 mm Glass

	<u>English</u>	<u>Metric</u>
Shading Coefficient	0.75	0.75
Relative Heat Gain (RHG)	155	488
Winter U-Value	0.32	1.79
Summer U-Value	0.33	1.88
%T _{solar} (IGU)	41	41
% R _{solar} (IGU)	15	15

Thermal data for the IGU are based on the following assumptions:

1. The coating is glazed on the #3 surface.
2. Both lites are 6-mm-thick clear glass.
3. The air gap is 0.5 inches (1.27 cm) and contains air.
4. %R_{solar} (IGU) is reflection to the outside of the building.
5. The units for RHG and U-Value are:

	<u>English</u>	<u>Metric</u>
RHG	BTU/hr-ft ²	W/m ²
U-Value	BTU/hr-ft ² -°F	W-m ² /°C

Table 8
Durability Tests Results

Test Type (Appendix 1)	Average Score	Std Dev
Humidity Test	9.8	0.18
Salt Fog Test	8.7	0.92
UV Exposure	9.9	0.12
Ammonium Hydroxide	9.6	0.47
Sodium Hydroxide	9.4	0.5
Hydrochloric Acid	9.3	0.59
Sulfuric Acid	9.9	0.11
Nitric Acid	9.9	0.06
Taber Abrasion	8.2	0.26

NOTE: Data taken from 8 Runs made in October 1992 on G-6
Samples Taken from Center of Lite
 $\text{Average Score} = (\text{Defect Score} + \text{Emmisivity Score}) / 2$

Table 9
Process Description
G-6 Development Coater Parameters

TiO₂

HRC-3000 Planar Cathode
90 % O₂ / 10 % Ar Gas Mixture
8 Passes
Source to Substrate = 3 " (7.6 cm)

40 kW Power
2.0 millitorr Pressure
Floating Anode

NiCrN_x

HRC-3000 Planar Cathode
60 % N₂ / 40 % Ar Gas Mixture
1 Pass, 3 inch Masking Slit
Source to Substrate = 3 " (7.6 cm)

6.4 kW Power
2.5 millitorr Pressure
Floating Anode

Ag

HRC-3000 Planar Cathode
60 % N₂ / 40 % Ar Gas Mixture
1 Pass
Source to Substrate = 5 " (12.7 cm)

8.9 kW Power
2.5 millitorr Pressure
Floating Anode

Si₃N₄

Dual C-MAG-3000 Planar Cathode
80 % N₂ / 15 % Ar / 5 % He Gas Mixture
2 Passes
Source to Substrate = 2.5 " (6.4 cm)
Magnets at 45 degree angle (Figure 1)

20.0 kW Power per Tube
4.0 millitorr Pressure
Floating Anode

End Shields on Targets

Table 10
Super-E™ IV Coating Design

<u>Layer</u>	<u>Thickness (Å)</u>	<u>DDR</u>	<u>Power Density</u> <u>Watts / mm</u>
TiO ₂	300	80	16
NiCrN _x	8	450	8
Ag	105	8000	8
NiCrN _x	8	450	8
Si ₃ N ₄	425	625	7.75

Table 11
Maximum Operating Powers

100 inch Substrate

<u>Layer</u>	<u>Cathode Racetrack (mm)</u>	<u>Cathode Type</u>	<u>Maximum Power (kW)</u>	<u># of Passes</u>
TiO ₂	5690	C-MAG-6000	91.0	1
NiCrN _x	6000	HRC-6000	48.0	1
Ag	6000	HRC-6000	48.0	1
Si ₃ N ₄	5690	C-MAG-6000	44.1	1

126 inch Substrate

TiO ₂	6800	C-MAG-7200	108.8	1
NiCrN _x	7200	HRC-7200	57.6	1
Ag	7200	HRC-7200	57.6	1
Si ₃ N ₄	6800	C-MAG-7200	55.8	1

Table 12
Coating System Solutions

Glass Size = 100" x 144"

<u>Layer</u>	<u># of Cathodes</u>	<u>Cycle Time (min : sec)</u>	<u>Power (kW)</u>	<u>Line Speed (IPM)</u>
<u>5 Coat Zone System</u>				
TiO ₂	8 Dual C-MAG	1:15	91.0	160
NiCrN _x	2 Planar		7.3	
Ag	1 Planar		5.4	
Si ₃ N ₄	3 Dual C-MAG		44.0	
<u>4 Coat Zone System</u>				
TiO ₂	6 Dual C-MAG	1:30	90.8	120
NiCrN _x	2 Planar		5.4	
Ag	1 Planar		4.0	
Si ₃ N ₄	3 Dual C-MAG		32.9	
<u>3 Coat Zone System</u>				
TiO ₂	3 Dual C-MAG	2:45	91.0	60
NiCrN _x	2 Planar		2.7	
Ag	1 Planar		2.0	
Si ₃ N ₄	2 Dual C-MAG		47	

Table 13
Coating System Solutions

Glass Size = 3.2 x 6.0 meter

Layer	# of Cathodes	Cycle Time (min : sec)	Power (kW)	Line Speed (cpm)
<u>5 Coat Zone System</u>				
TiO ₂	9 Dual C-MAG	2:00	97.4	410
NiCrN _x	2 Planar		8.8	
Ag	1 Planar		6.5	
Si ₃ N ₄	3 Dual C-MAG		53.0	
<u>4 Coat Zone System</u>				
TiO ₂	6 Dual C-MAG	2:30	107.9	300
NiCrN _x	2 Planar		6.5	
Ag	1 Planar		4.8	
Si ₃ N ₄	3 Dual C-MAG		39.1	
<u>3 Coat Zone System</u>				
TiO ₂	3 Dual C-MAG	4:15	108.7	150
NiCrN _x	2 Planar		3.3	
Ag	1 Planar		2.4	
Si ₃ N ₄	2 Dual C-MAG		29.6	

Super-E IV Optical Performance

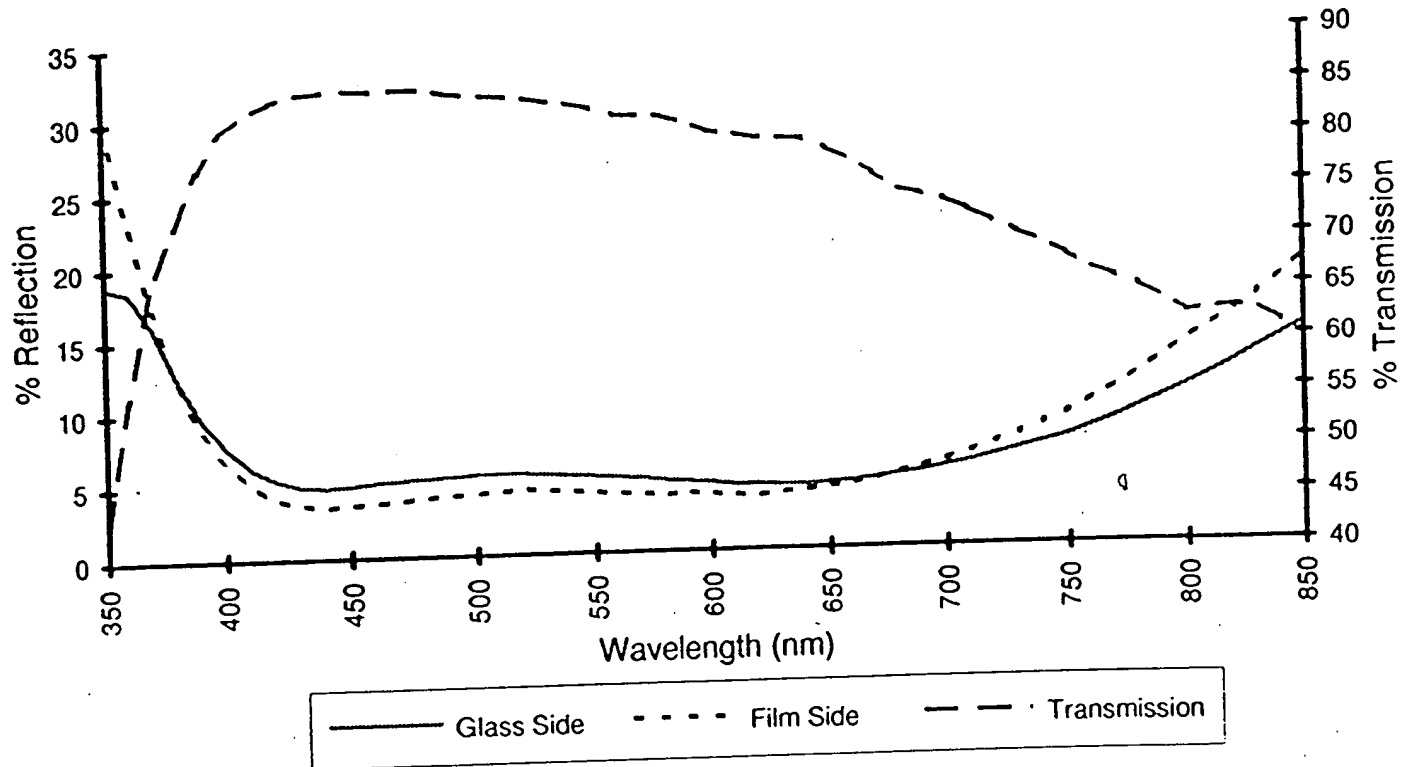
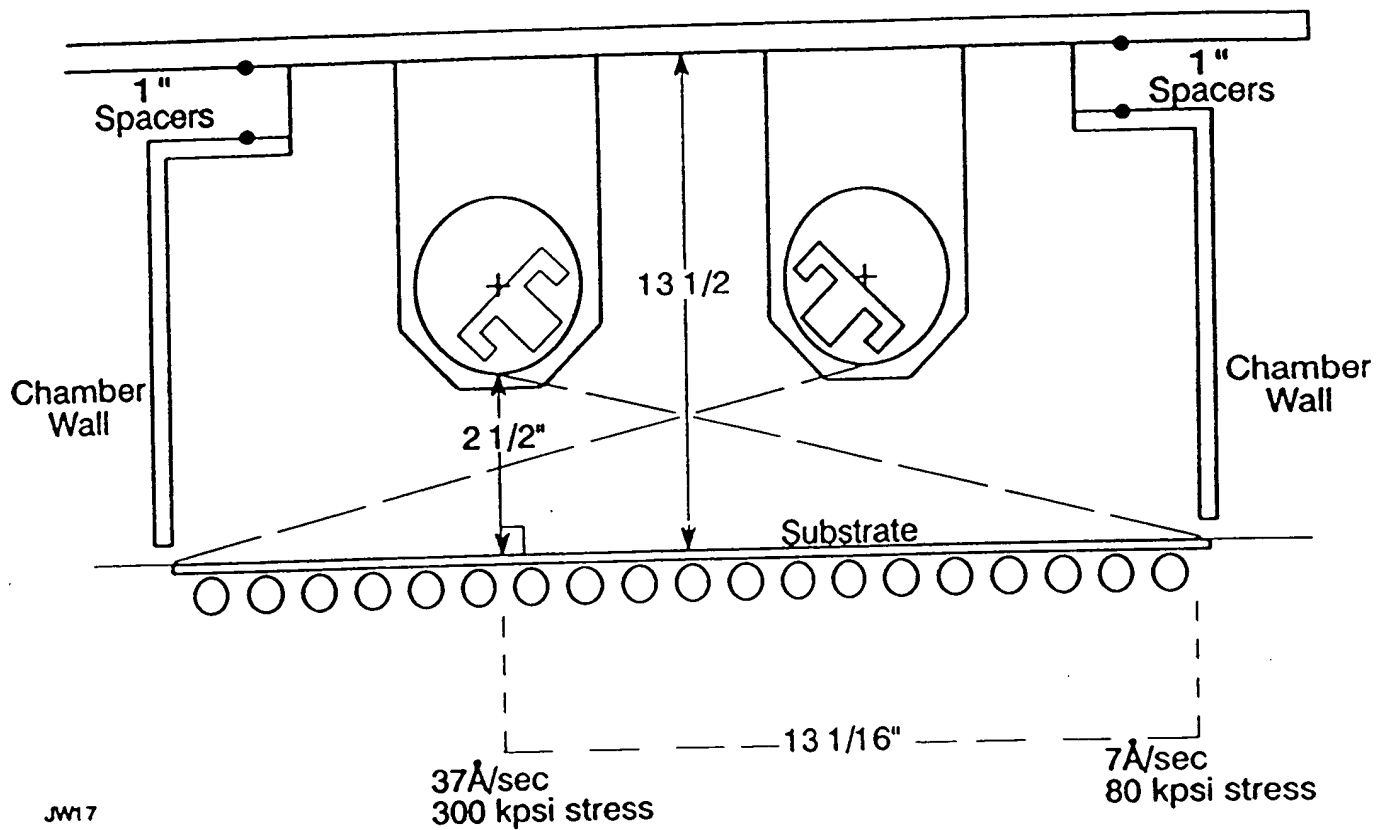


Figure 1

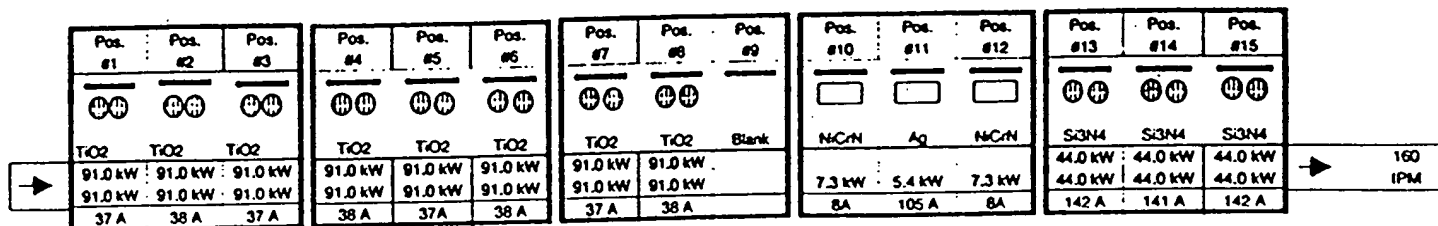
Dual C-MAG® with Magnets at 45° Machine - G-6



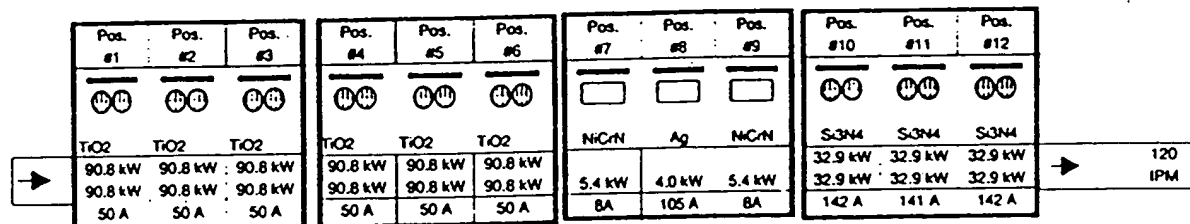
JW17

Figure 2

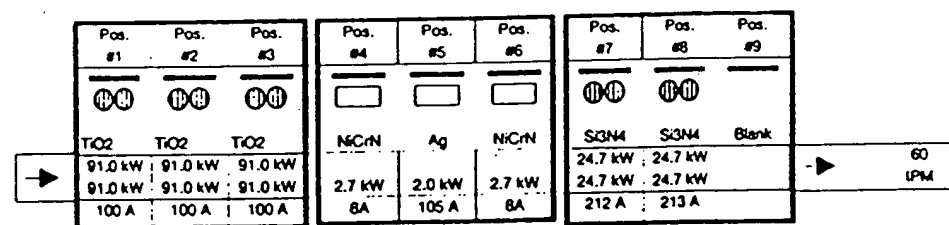
Super-E IV Cycle Time / System Configuration for 144 inch Glass



5 Coat Zone
CYCLE TIME = 1 min 15 sec



4 Coat Zone
CYCLE TIME = 1 min 30 sec



3 Coat Zone
CYCLE TIME = 2 min 45 sec

Cathode Type - 6000 Series

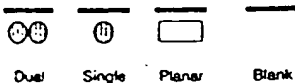


Figure 3

Super-E IV Cycle Time / System Configuration for 6 meter Glass

Pos. #1	Pos. #2	Pos. #3	Pos. #4	Pos. #5	Pos. #6	Pos. #7	Pos. #8	Pos. #9	Pos. #10	Pos. #11	Pos. #12	Pos. #13	Pos. #14	Pos. #15	
TiO2	TiO2	TiO2	TiO2	TiO2	TiO2	TiO2	TiO2	TiO2	NiCrN	Ag	NiCrN	S3N4	S3N4	S3N4	
97.4 kW	97.4 kW	97.4 kW	97.4 kW	97.4 kW	97.4 kW	97.4 kW	97.4 kW	97.4 kW	8.8 kW	6.5 kW	8.8 kW	53.0 kW	53.0 kW	53.0 kW	410 cpm
97.4 kW	97.4 kW	97.4 kW	97.4 kW	97.4 kW	97.4 kW	97.4 kW	97.4 kW	97.4 kW	8.8 kW	6.5 kW	8.8 kW	53.0 kW	53.0 kW	53.0 kW	
33 A	34 A	33 A	33 A	34 A	33 A	33 A	34 A	33 A	8A	105 A	8A	142 A	141 A	142 A	

5 Coat Zone
CYCLE TIME = 2 min 00 sec

Pos. #1	Pos. #2	Pos. #3	Pos. #4	Pos. #5	Pos. #6	Pos. #7	Pos. #8	Pos. #9	Pos. #10	Pos. #11	Pos. #12	
TiO2	TiO2	TiO2	TiO2	TiO2	TiO2	NiCrN	Ag	NiCrN	S3N4	S3N4	S3N4	
107.9 kW	107.9 kW	107.9 kW	107.9 kW	107.9 kW	107.9 kW	6.5 kW	4.8 kW	6.5 kW	39.1kW	39.1kW	39.1kW	300 cpm
107.9 kW	107.9 kW	107.9 kW	107.9 kW	107.9 kW	107.9 kW	6.5 kW	4.8 kW	6.5 kW	39.1kW	39.1kW	39.1kW	
50 A	50 A	50 A	50 A	50 A	50 A	8A	105 A	8A	142 A	141 A	142 A	

4 Coat Zone
CYCLE TIME = 2 min 30 sec

Pos. #1	Pos. #2	Pos. #3	Pos. #4	Pos. #5	Pos. #6	Pos. #7	Pos. #8	Pos. #9	
TiO2	TiO2	TiO2	NiCrN	Ag	NiCrN	S3N4	S3N4	Blank	
108.7 kW	108.7 kW	108.7 kW	3.3 kW	2.4 kW	3.3 kW	29.6 kW	29.6 kW		150 cpm
108.7 kW	108.7 kW	108.7 kW	3.3 kW	2.4 kW	3.3 kW	29.6 kW	29.6 kW		
100 A	100 A	100 A	8A	105 A	8A	212 A	213 A		

3 Coat Zone
CYCLE TIME = 4 min 15 sec

Cathode Type - 7200 Series

Dual
 Single
 Planar
 Blank

Figure 4



Super -E III™

The Super-E III™ coating consists of five layers in the following order:

glass / Si₃N₄ / NiCr / Ag / NiCr / Si₃N₄ / air

Typical optical data for the coating are as follows:

III. "C" %Transmission: 76 ± 1

%Reflection (Film): 4 ± 1

"a": -1 ± 1

"b": -6 ± 1.5

%Reflection (Glass): 8 ± 1

"a": -2.5 ± 1

"b": -7.5 ± 1.5

Solar %Transmission: 62 ± *

%Reflection (film): 14 ± *

%Reflection (glass): 12 ± *

Emissivity 0.16 or less

*No proposed error bars for these specifications.

Typical power settings for the nickel chromium (NiCr) and silver (Ag) are much lower than those for the silicon nitride (Si₃N₄). An example: for samples run on Airco's one meter wide, flat glass development coater, power, and line speed data were as follows:

Si₃N₄ Power = 36 kW with one dual C-MAG® 3000 rotatable cathode

Line speed (base) = 63 IPM (27 mm/sec) × 1 pass

Line speed (top) = 45 IPM (19 mm/sec) × 1 pass

Ag Power = 3.5 kW with one HRC-3000 planar cathode

Line speed = 293 IPM (124 mm/sec) × 1 pass

NiCr Power = 0.95 kW with one HRC-3000 planar cathode

Line speed = 293 IPM (124 mm/sec) × 1 pass

Super-E III

The general tuning sequence is as follows: first, using clear glass sample lites, approximate power and line speed settings are determined separately for the two silicon nitride layers and for the silver layer. The first Si_3N_4 layer is sputtered onto the bare glass, then the first nickel chromium layer is deposited on top of that. The silver is laid down and tuned for emissivity, then the NiCr thickness is tested with an adhesion snap tape test. After that comes another NiCr layer, then the top silicon nitride layer. After that another adhesion tape test is made to test the top silicon nitride's adhesion. Individual sample lites should be run separately for all three materials first in order to get approximate values for power and line speed before tuning of the entire stack begins. The details follow.

The thickness of the two silicon nitride layers may be estimated using a Spectrogard™ spectrophotometer and the attached graph (Figure 1), which shows the change in transmission and transmitted Hunter "b" value color variation with film thickness. For example, the first Si_3N_4 layer, nominally 325 Å thick, should allow about 82% light transmission when coated on a three millimeter (1/8 inch) glass substrate, while the "b" color value ought to be about +2.8. Thus, the first nitride layer is tuned by varying the power and/or line speed to obtain these two values. Note that the transmission can be 82% for two thicknesses, 325 Å or 1060 Å, but the "b" color at 1060 Å thickness will be much lower (somewhere near -4), so one can tune the nitride with certainty for 325 Å thickness by getting both transmission and "b" color value correct. Power and line speed settings for the top nitride should be set initially in the same way on bare glass. That is, sample lites should be run to obtain an approximate thickness of 450 Å, i.e. transmission about 78% and "b" color about +2.9. Then the power and line speed should be noted for later reference when running the complete stack.

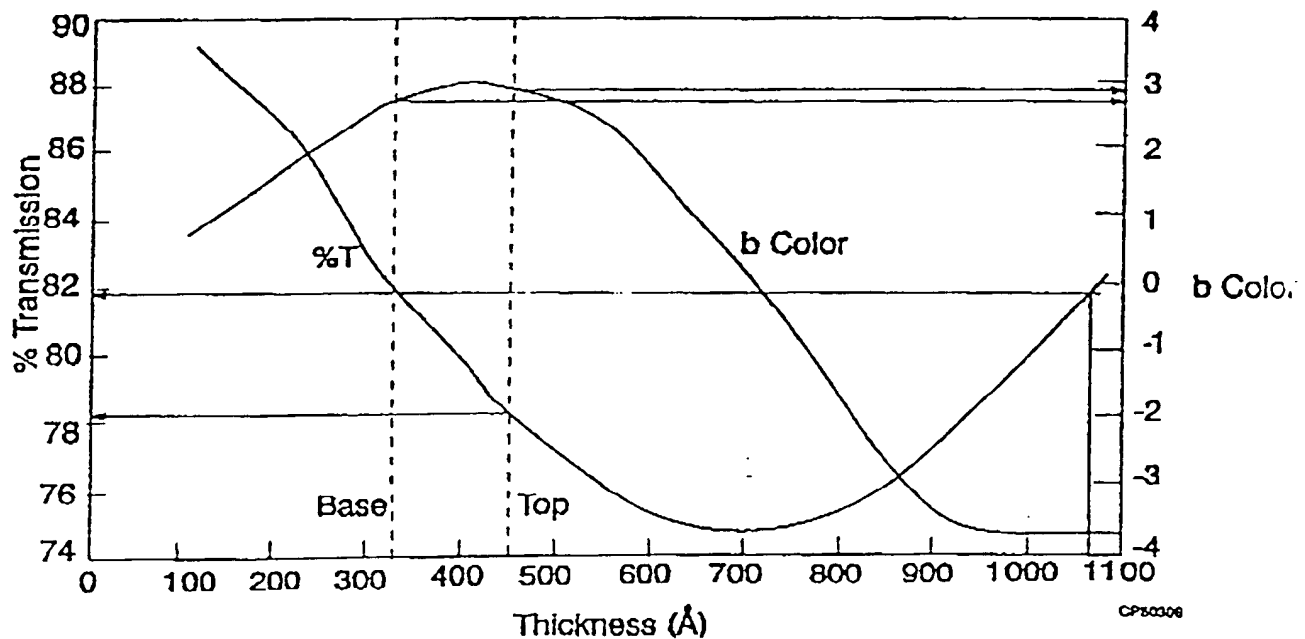


Figure 1 - Si_3N_4 on 3mm Glass

Since the overall production efficiency will be limited by the two relatively thick Si_3N_4 layers, it is important that the power and line speeds for these layers be as high as possible without melting or otherwise damaging the targets (as with extreme arcing). Let's take an example: by running sample lites, we have found out that a power setting of 18 kW and a line speed of 31 IPM will give the right thickness for the base silicon nitride layer. We can then increase the power and line speed if we keep the ratio of power/line speed constant, thereby maintaining the same thickness. So if we increase the power by, say, twenty percent (from 18 kW to 21.6 kW), we must also increase the line speed by twenty percent (from 31 IPM to 37 IPM). We keep increasing the power and line speed, keeping their ratio constant, until the arc control/arc diverter assemblies can no longer suppress the arcing, at which time it is prudent to reduce the power to a level a bit under the point where the arcing stops. The line speed must then be reduced proportionately. These power and line speed settings are then very close to the right ones to use for production. We will then "fine-tune" the coating for the correct transmission and color specifications, to be discussed below.

Bare silver samples on three millimeter (1/8 inch) glass are initially tuned to a transmittance of $60 \pm 2\%$ to get approximately correct power and line speed values, since the entire stack will later be "fine tuned" by varying the silver layer thickness to obtain an emissivity of 0.16 or less using either an emissometer or an infrared (IR) spectrophotometer. An alternative is to use a four-point probe to measure the stack's sheet resistance, which should be 15 ohms/square. The sheet resistance is also measured on the whole stack.

The first nickel chromium layer is deposited on the Si_3N_4 base and tuned to be as thin as possible while still maintaining a uniform layer for the overlying silver layer to adhere to. Silver will not adhere directly to silicon nitride, thus the need for the adhesion layer of NiCr. The NiCr should be just thick enough to be able to pass an adhesion snap tape test. A power setting of about one-third that for the silver is a good place to start the NiCr tuning, assuming the line speeds are the same. If the NiCr is too thick, the transmission will be too low, and if it's too thin, it will fail the adhesion snap tape test. That is, the coating will end up on the tape rather than on the glass. See Section 6.3.8 ("Adhesion Tape Test") in Chapter 6, Quality Control, for details about this test.

Using the power and line speed settings determined from running silver sample lites on bare glass (see above) as a starting point, we can then lay down the silver to the correct thickness on top of the first NiCr layer. As stated above, tune the silver layer for emissivity.

The second NiCr layer is also an adhesion and barrier layer. That is, it provides a layer to which the top silicon nitride layer can adhere, and it separates the silver layer from that top nitride layer. If the upper NiCr layer is too thin, it will not act to separate the silicon nitride from the underlying silver. The silicon nitride won't adhere to the silver in that case, and the coating will fail the snap tape test. In addition, some of the silver will react with available nitrogen in the Si_3N_4 layer, forming silver nitride and increasing the emissivity of the coating. The top NiCr layer should therefore be as thin as possible without allowing the silver to come into

Super-E III

direct contact with the nearby Si_3N_4 . The same adhesion tape test is used to test the top NiCr as was used on the bottom NiCr. Again, as with the bottom NiCr layer, making the top NiCr too thick will decrease the transmission. The two layers are usually adjusted together, and fine control of their thicknesses is critical. They are nominally about 7 Å thick.

Glass Side Color Tuning

Production personnel will have to color tune Super-E III™ for both glass and film side color at the same time. That is, changes in the thicknesses of the two silicon nitride layers will affect the reflected color on both sides of the glass. However, since the tuning of the glass side color is a little easier to understand initially than that of the film side, we will start with glass side color tuning.

The top layer of silicon nitride is usually tuned together with the bottom layer. Figure 2 shows how varying the two silicon nitride layers' thicknesses affects the Hunter "a" color value in glass side reflection. Lines of constant "a" value are plotted, much like lines of constant height on a contour map. Due to the scaling of the graph, it appears that the base silicon nitride layer is very insensitive in "a" value for thickness changes compared to the top layer, but such is not the case. Let's take an example in the middle of the chart: the "a" color for the nominal thickness values is $a = -1.89$. (Note: as these are color values generated by a computer program, they don't agree precisely with those obtained in producing Super-E III in real life. However, the computer does model the changes in color with thickness changes very well, and that's what we're trying to find out.) Now make the same size change, first to the base nitride layer, then

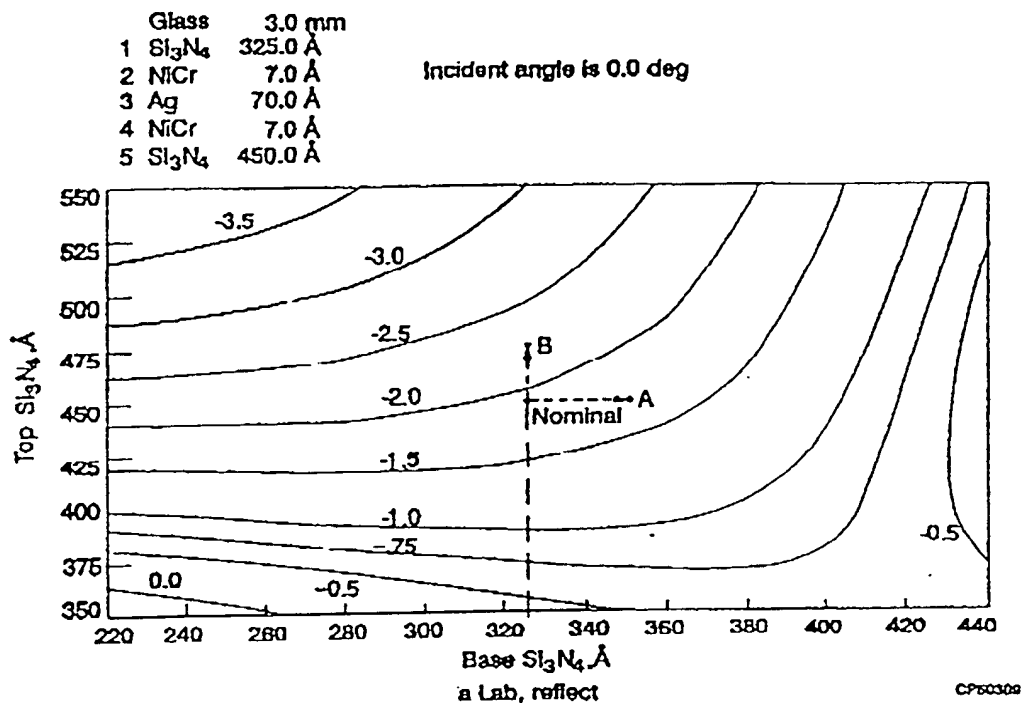


Figure 2 - Glass Side Color Tuning the Si_3N_4 Layers: L a b "a" Map

to the top one. Point A in Figure 2 shows the "a" color for a 25 Å increase in thickness for the base silicon nitride. The color changes from -1.89 to -1.71, a change of + 0.17. For the same thickness change for the top layer, look at point B. The change is from -1.89 to -2.20, a change of -0.31.

There are two lessons to be learned here: 1) the top nitride layer affects "a" color more than the bottom layer does, although the rates of change of color are somewhat comparable, and 2) the direction of the color changes are opposite. That is, for the base layer, the color varies directly with the thickness ("a" goes up as the thickness increases). For the top layer, the color varies inversely ("a" goes down as the thickness increases).

Tuning the nitride layers for "b" color in glass side reflection is more difficult, although it follows the same strategy as outlined above for the "a" value. Of course, thickness changes will affect both "a" and "b" values at the same time. Look at Figure 3. Starting with the nominal thicknesses (Point D), one can see that making either layer thicker usually moves the "b" value more positive.

There are two exceptions to this rule. The large area where the two -6.0 contour lines converge in the upper right part of Figure 3 is one of them. Here a three-dimensional plot (not shown) would indicate a nearly flat area, a saddle-shaped area running diagonally to the upper right. In this area the "b" value changes very slowly with thickness, and its direction reverses. It would be very difficult to tell where one is with only this map as a guide, but by referring to the Lab "a" map, Figure 2, at the same time, one's location can be easily pinpointed. The other exception is the lower center and lower left corner areas, where the top layer is substantially

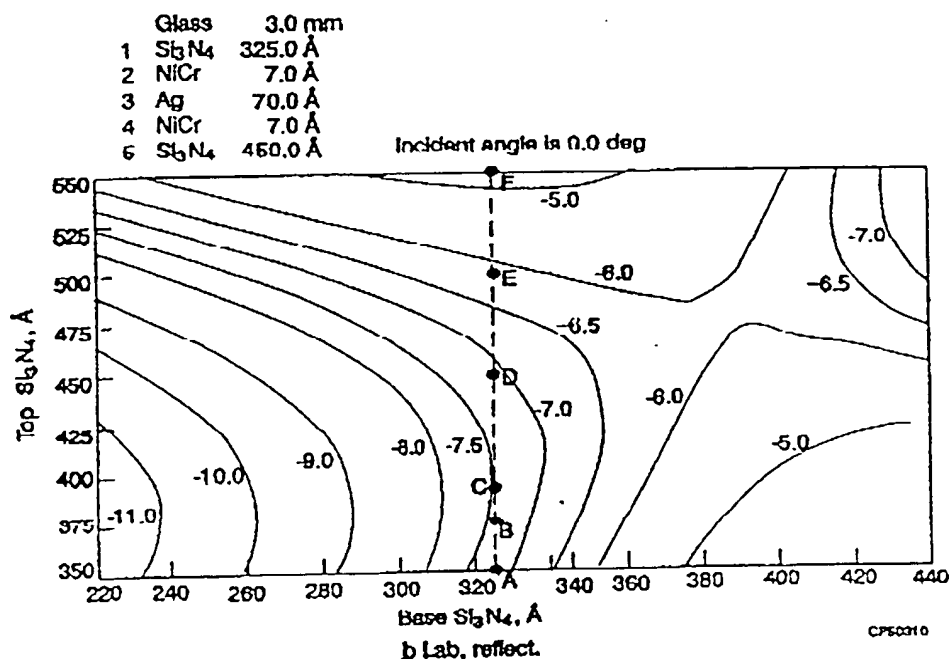


Figure 3 - Glass Side Color Tuning the Si_3N_4 Layers: L a b "b" Map

Super-E III

less than the nominal thickness. Here making the top nitride thicker sometimes moves the "b" value more negative. In fact, if the bottom layer has already been tuned in to the nominal thickness of 325 Å (using Figure 1 and the rules given above), one can see the "b" value reverse as the top nitride layer is thickened. Refer to Figure 3 or 4, where a cross-section (A-F) has been drawn at the nominal thickness for the base silicon nitride layer. At 350 Å top layer thickness the "b" color is -7.19 (Point A). Then it drops to -7.41 at 375 Å thickness (Point B) and continues to decrease until it reaches a minimum of -7.49 at about 392 Å (Point C). Then it starts to increase until its value is -7.12 at the nominal thickness of 450 Å (Point D). The "b" value then continues to increase for increasing top layer thickness: at 500 Å (Point E) it is -6.13, and at 550 Å (Point F) it is -4.78.

The way to tune Super-E III for final color in glass side reflection is therefore the following: use the previously given instructions for transmittance and transmitted "b" color to get the two silicon nitride layers close to their proper thicknesses, then fine-tune the entire coating's "a" and "b" glass side color by adjusting the thicknesses of the top and bottom layers, taking care to watch both color values. By using Figures 2 and 3, it should be fairly easy to tune the coating for glass side color.

The easiest way to tune the two nitride layers together is to make a transparent copy of one of

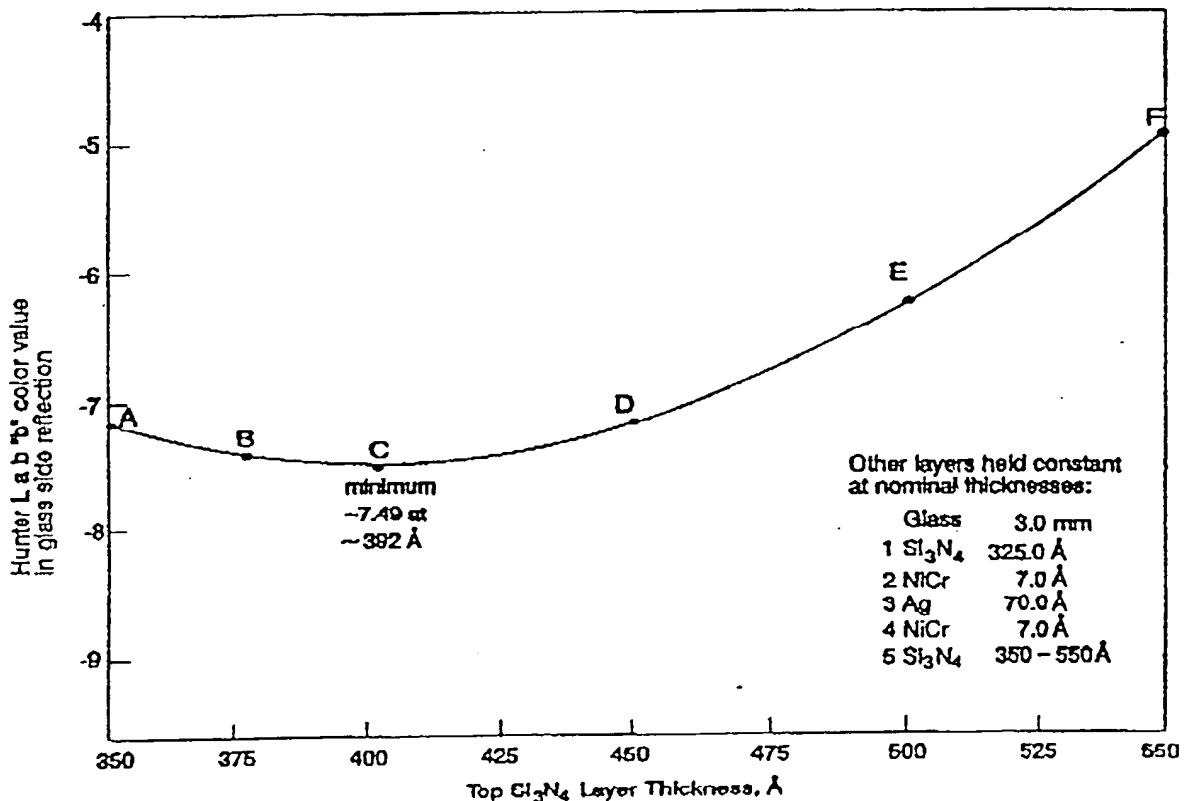


Figure 4 - Glass Side Hunter L a b "b" Color Change vs. Top Si_3N_4 Thickness

CP60311

these "color contour maps" and use it as an overlay over the other one. After aligning the two charts properly, we only have to find the intersection of the specified "a" and "b" color value contour lines to see where we want the coating to be. For example, let's take our nominal thicknesses, 325 Å base and 450 Å top, as a good compromise for both glass and film side color. We first plot the measured glass side color values for a sample coating, say, for example, "a" = -2.0 and "b" = -9.00. This occurs only for thicknesses of about 270 Å (base) and about 440 Å (top). See Figures 2 and 3. Therefore we would have to make the base layer somewhat thicker ($325 \text{ Å} - 270 \text{ Å} = 55 \text{ Å}$) and the top layer just a little thicker ($450 \text{ Å} - 440 \text{ Å} = 10 \text{ Å}$). A sample is run at new power and line speed settings; the glass side color values for the new sample are measured, and the charts are again consulted to plot how close to "spec" the glass side reflected color now is. Remember, though, we will be looking at the film side color, too, when we change the nitride layers' thicknesses. We continue to make the silicon nitride layers thicker or thinner (or one thicker and the other thinner, as the case demands), getting ever closer to the color specifications. This "dialing in" or "tuning" loop continues until we are satisfied that the glass side color is right. A little experience adjusting power and line speed settings will enable the operator to do this fairly easily after a short time.

Film Side Color

Tuning the coating for film side color is done in exactly the same way as for glass side color. There are two differences, though: 1) the coating is more sensitive in "b" value for film side color (that is, the film side color can "go blue" rather quickly for small changes in silicon nitride thickness changes), and 2) the color specifications are quite different. Of course, changing either silicon nitride layer will affect both film side and glass side color values. Therefore, all four color values must be monitored when adjusting the silicon nitride layer thicknesses. In general, film side color is the color specification used for on-line color control. However, either glass side or film side color may be the final criterion for quality control, depending on how the coating will be finally glazed. That is, if the coating will end up on the number three surface (counting the glass surfaces from the outside in) of an insulated glass unit (IGU), then the film side color will usually be the more important specification. If the coating is to go on the number two surface of the IGU, then the glass side color will be critical.

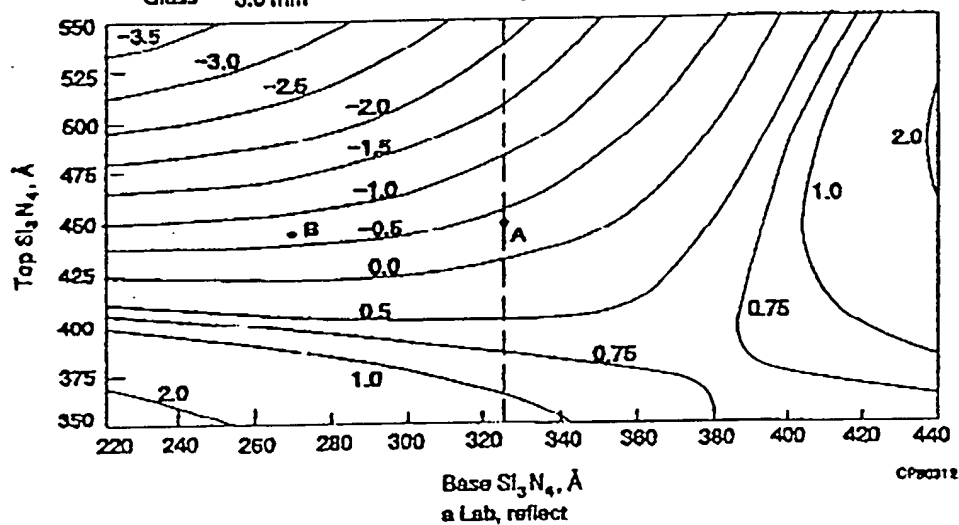
Figures 5 and 6 map "a" and "b" color for film side reflectance. Notice that the contour lines for the Airco specification, "a" = -1 and "b" = -6, do not intersect anywhere on the chart! But notice also that the two lines are close together at the nominal thicknesses of 325 Å base and 450 Å top layer (Point A in Figure 5). In fact, at Point A, "a" = -0.31 and "b" = -6.19. These values are well within the \pm one color unit of the specification.

Let's look at the same beginning sample that we did previously in the glass side tuning section. There we determined from the glass side color that the nitride layers were about 270 Å and 440 Å thick for the base and top, respectively. If we plot those thicknesses on Figures 5 and 6,

Super-E III

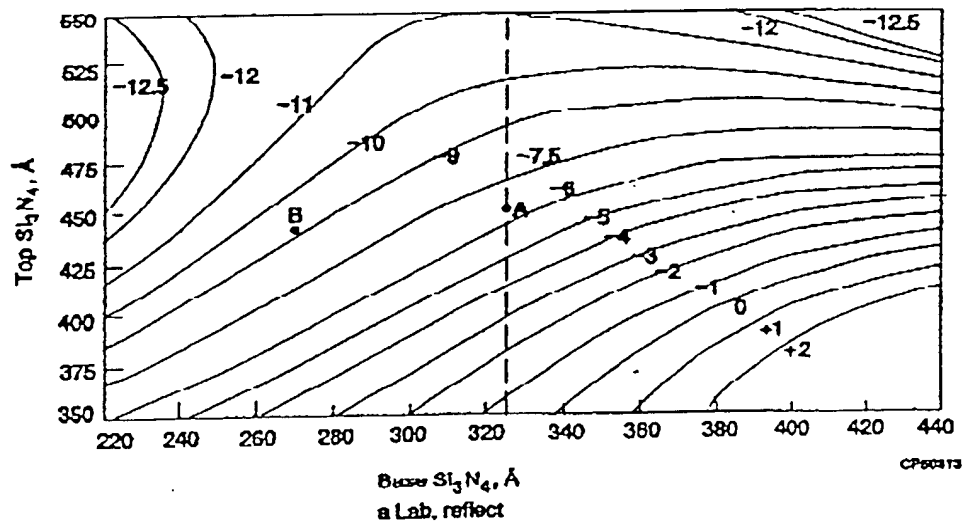
Si_3N_4 450.0 Å
 NiCr 7.0 Å
 Ag 70.0 Å
 NiCr 7.0 Å
 Si_3N_4 325.0 Å
 Glass 3.0 mm

The incident angle is 0.0 deg.

Figure 5 - Film Side Color Tuning the Si_3N_4 Layers: L a b "a" map

Si_3N_4 450.0 Å
 NiCr 7.0 Å
 Ag 70.0 Å
 NiCr 7.0 Å
 Si_3N_4 325.0 Å
 Glass 3.0 mm

The incident angle is 0.0 deg.

Figure 6 - Film Side Color Tuning the Si_3N_4 Layers: L a b "b" map

Si_3N_4 450.0 Å
 NiCr 7.0 Å
 Ag 70.0 Å
 NiCr 7.0 Å
 Si_3N_4 325.0 Å
 Glass 3.0 mm

The Incident angle is 0.0 deg.

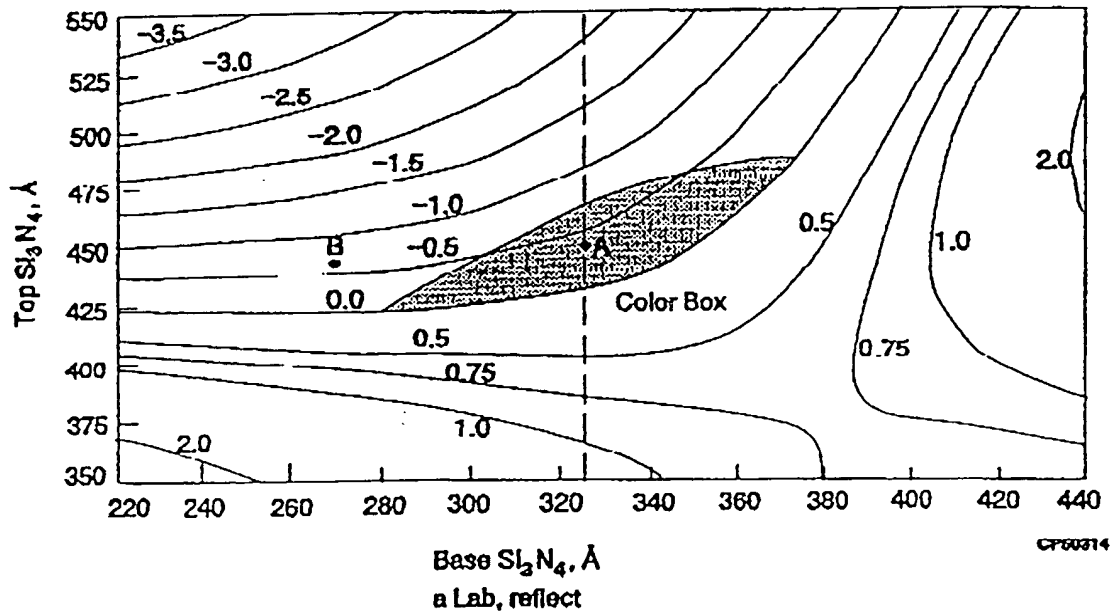
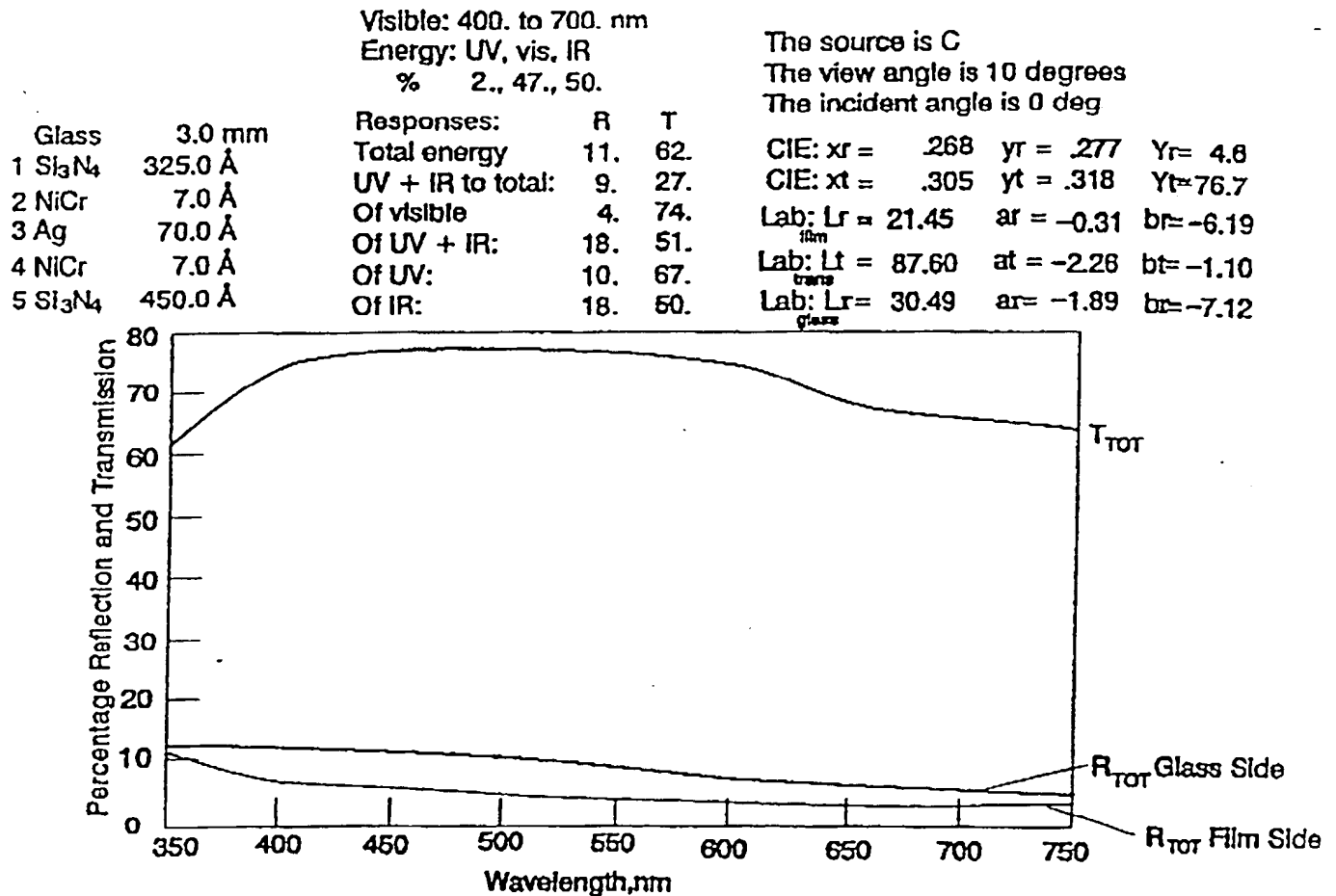


Figure 7 - Color Box For Tuning Si_3N_4 Layers

we find Point B, where the film side color values are about "a" = -0.55 and "b" = -9.10. The "a" value is within one unit of the specification, but not the "b" value. Again, just as with the glass side color, we will have to adjust the nitride layers' thicknesses to get the color values within the specification. That is, we would want to make the base considerably thicker (325 Å - 270 Å = 55 Å) and the top layer just a little thicker (450 Å - 440 Å = 10 Å). We adjust the thicknesses of the two nitride layers to work towards Point A, where all four color values are well within the error bars of the specifications.

Figure 7 shows a 'color box' for all four color numbers for Super-E III™ superimposed on the film side "a" contour lines. Any coating falling within the "box" will satisfy the Airco specifications for color within the given error bars. Notice that the nominal thicknesses (at Point A) produce a coating very nearly in the center of the "color box".

Figure 8 shows typical values for color, transmission, and reflection at the nominal thicknesses. It should only be necessary to make very small changes in the nitride layer thicknesses during production to adjust the coating for small changes in color. Transmission and emissivity can be maintained within specification by changing the nickel chromium and silver thicknesses, respectively, by small increments. Of course, when thinning out the NiCr layer, one should

Super-E III

CP50315

Figure 8 - Super-E III Nominal Thicknesses

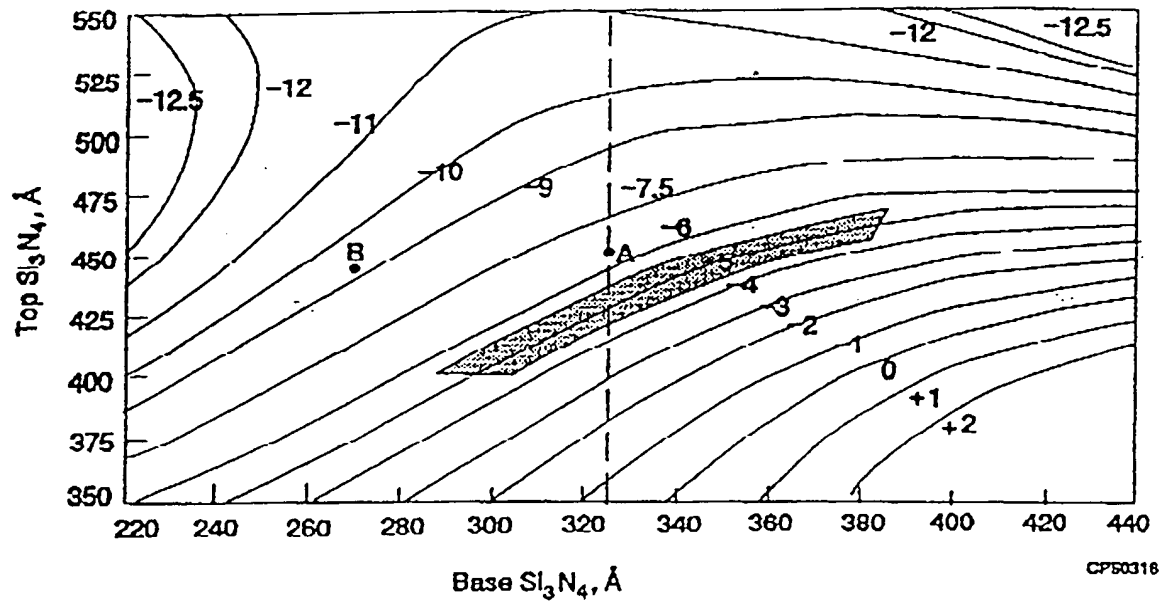
check for adhesion with the snap tape test. If the silver thickness is changed, the emissivity will need to be re-tested, too. Also, be aware that adjustment to the silver layer will change the color of the coating: making the silver thicker will make the glass side color bluer ("b" more negative) and the film side color redder ("a" more positive). In production the silver and nickel chromium layers (especially the latter) will probably require more "tweaking" than the silicon nitrides.

In the re-tuning the Super-E III coating after a vent, one should use the most recent settings that gave good results, then make small adjustments using the procedure outlined above. A final warning: experience has shown that the low emissivity coatings, including Super-E III, are very susceptible to contamination, especially leaks. An air or water leak will dramatically affect color. After a vent, it will take the coater some time to return to a stable condition. The time interval can range from a few hours up to two days depending on the condition of the coater and the length of the vent. It is very important to monitor the Super-E III coating during

Si_3N_4 450.0 Å
 NiCr 7.0 Å
 Ag 70.0 Å
 NiCr 7.0 Å
 Si_3N_4 325.0 Å
 Glass 3.0 mm

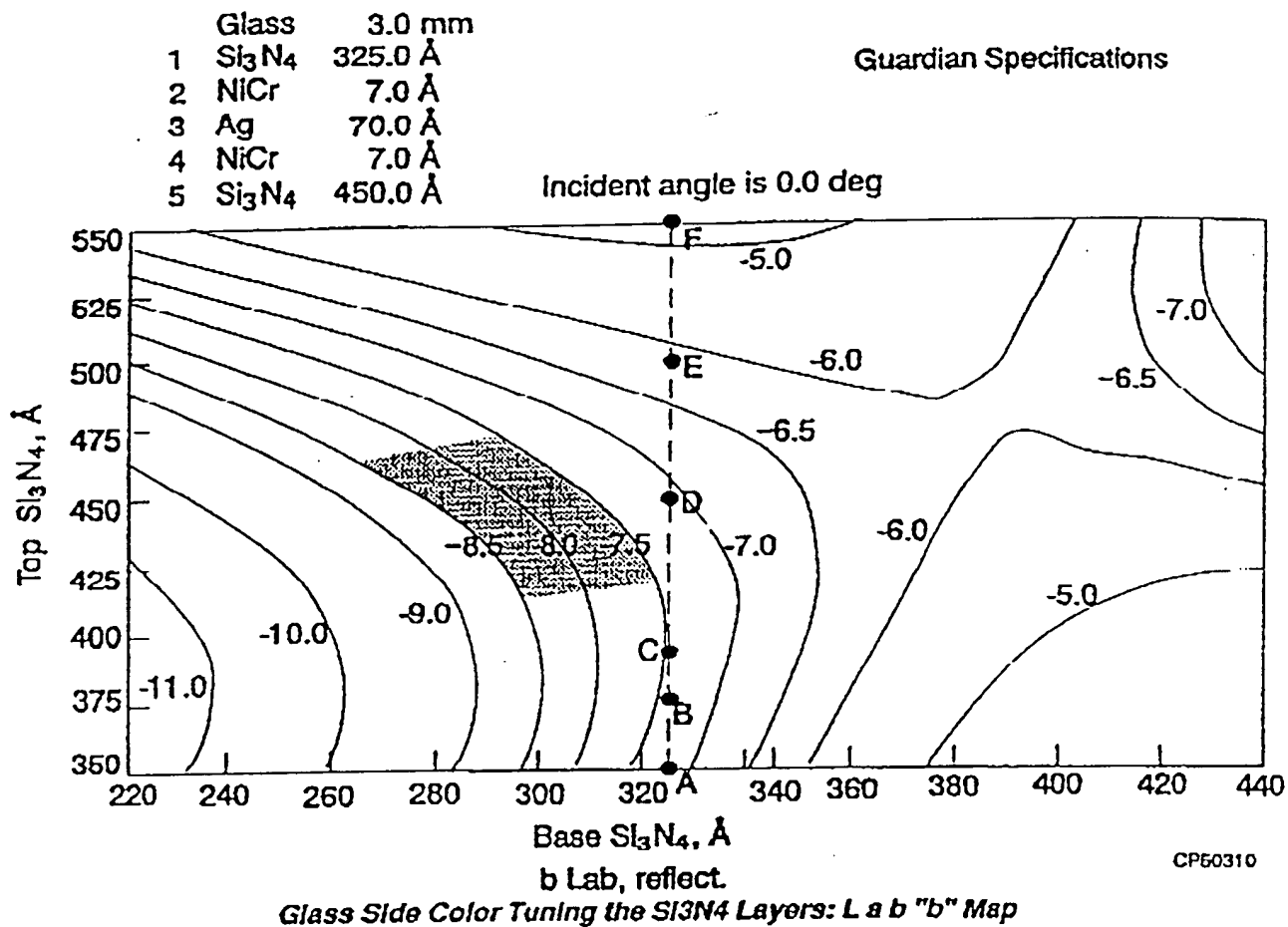
Guardian Specifications

The Incident angle is 0.0 deg.



Base Si_3N_4 , Å
 a Lab, reflect
 Film Side Color Tuning the Si_3N_4 Layers: L a b "b" Map

this time of instability. At such a time the product should be closely watched for any changes in optical or physical properties.

Super-E III

Super-E III

Airco vs. Guardian

<u>Airco</u>		<u>Guardian</u>
76 ± 1	%T	$76 \pm .5$
4 ± 1	%Rf	$5 \pm .5$
-1 ± 1	a	$0 \pm .5$
-6 ± 1.5	b	$-5 \pm .5$
8 ± 1	%Rg	$8 \pm .5$
-2.5 ± 1	a	$-2 \pm .5$
-7.5 ± 1.5	b	$-8 \pm .5$



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P.O. Box 2529
Fairfield, California 94533-0252
Telephone: 707-423-2100
TLX: 4990342
FAX: 707-425-7889

Cather,

Please send copies to

- Klaus Hartig
- Ray Nalepa
- Karl Straky (Phil Leigle)
- Ken Haveren

Thanks,
Ref

January 15, 1991

Mr. Rex Tapp
GUARDIAN INDUSTRIES
43043 West Nine Mile Road
Northville, MI 48167

RE: Super-E IIITM Low-E Coating

Last October we sent you samples of our new Super-E III Low-E coating for your evaluation. The response we received from our licensees indicated an extremely strong interest in the product, and we are now pleased to officially release the coating for your production consideration.

As mentioned in our previous correspondence, this product is made possible by the ability to use the C-MAGTM rotatable cathode to deposit silicon in the form of Si₃N₄. The product is extremely durable and has the possibility of being fabricated into insulating glass without the need to delete the coating from the edge of the glass. Due to various available sealants and fabrication techniques, each licensee will need to make their own evaluation of this possibility.

Please contact this office if you desire to receive an additional 12" x 12" (305mm x 305mm) sample of this product. Our Technology Group is available to respond to any detailed questions you may have regarding this release.

Sincerely,

Donoho S. Hall
Vice President
Marketing & Sales
11911

AIRCO SOLAR PROCESSES CONFIDENTIAL. THIS INFORMATION IS SUBJECT TO THE
CONFIDENCE OBLIGATION SET FORTH IN THE AGREEMENT DATED OCT. 1, 1982
BETWEEN GUARDIAN INDUSTRIES CORP. AND AIRCO, INC.

SUPER-E IIITM COATING PROCESS RELEASE

A. Introduction

A more durable form of Low-E film has been developed. Compared to the previous standard, Super-E IITM coating, this Super-E IIITM film has increased resistance to abrasion, humidity and various corrosive agents. This has been accomplished with the use of the technology made possible by the C-MAGTM rotatable cathode. The Super-E III coating uses Si_3N_4 in place of the oxide layer of the Super-E II coating in addition to a new, innovative barrier layer, resulting in a far more durable film stack.

The following coating release details the coating stack design, optical specifications, durability test results and process cycle time estimates.

B. Coating Design

The stack design for the Super-E III coating is:

Glass | Si_3N_4 | NiCr | Ag | NiCr | Si_3N_4
 3mm | -325Å | Barrier | -100Å | Barrier | -450Å

C. Optical Performance

Table I shows optical measurements averaged on six samples of the Super-E III coating made in Airco's flat glass development coater, G-6. Thermal calculations are shown in Table II.

D. Process Data

Typical production set up information is shown in Table III. Please note that precise control of the NiCr barrier layer thickness is required. The low power required to deposit the thin barrier layers require a power supply capable of precise control in the 0-5 kW range. Systems capable of a 60 second cycle time will require three dual C-MAG cathodes, fitted with Si targets for each Si_3N_4 layer.

A starting point for the nitride layer depositions can be obtained by using the approximate physical thicknesses noted above and the following deposition rate data:

$$\text{Si}_3\text{N}_4 \text{ dynamic deposition rate} = 700 \text{ Å} \cdot \text{mm}^2 / \text{joule}$$

To calculate the nominal thickness of film deposited with a given cathode, line speed and power, the following equation is used:

$$\text{Thickness (Å)} = \frac{\text{DDR (Å} \cdot \text{mm}^2 / \text{joule)} \cdot \text{Power (W)} \cdot \text{N (\# Cathodes)}}{\text{Line speed (mm/sec)} \cdot \text{Racetrack Length (mm)}}$$

NOTE: The racetrack length in mm is typically indicated in the cathode nomenclature; for example, an HRC-6000 cathode has a nominal racetrack length of 6000 mm.

The thickness of the silicon nitride may also be estimated by using a Spectrogard and the attached graph. Graph I shows the variation of the transmission and the transmitted 'b' color with change in film thickness. For example, a layer of Si_3N_4 with a transmission of 75% could either be 625 or 775 angstroms thick. By noting where the %T 'b' color value falls, the correct thickness could be estimated.

E. Durability Data

Durability testing included an abrasion test and several corrosion tests. Corrosion defects were looked for after humidity, salt fog, salt dot and UV exposures. A new test simulates typical in-service cleaning by soaking the sample in ammonium hydroxide. Test results are given in Table IV, with the test descriptions listed in the appendix. Test results indicate that the Super-E III coating is greatly improved in durability over the Super-E coating and over the Super-E II coating with its TiO_2 overcoat. These test data indicate that the Super-E III coating may have durability sufficient to use in a non-sealed, double-glazed unit. However, the Super-E III coating is not recommended for use in monolithic applications.

F. Conclusions

The Super-E III coating offers a significant improvement in durability over earlier films, while maintaining a high standard of aesthetic quality and functional performance.

Table I
Super-E III Coating Optical Data

		<u>Average +/- S.D.</u>	<u>Proposed Spec</u>
III. 'C'	%T	75.0 +/- 0.3	76 ± 1
	%Rf	4.3 +/- 0.1	4 ± 1
	'a'	-0.3 +/- 0.4	-1 ± 1
	'b'	-5.5 +/- 0.4	-6 ± 1.5
	%Rg	8.4 +/- 0.3	8 ± 1
	'a'	-2.4 +/- 0.3	-2.5 ± 1
	'b'	-8.0 +/- 0.6	-7.5 ± 1.5
Solar	%T	61.9 +/- 0.4	
	%Rf	13.9 +/- 0.6	
	%Rg	12.4 +/- 0.4	
	Emissivity	0.16 +/- 0.01	less than 0.16

Emissivity is normal emissivity based on specular reflectance measurements between 5-15 microns.

The data are the average of six samples.

Table II
Super-E III Coating Thermal Data

IGU Thermal Data

English

Average +/- S.D.

U-Value	Winter	0.39 +/- 0.0
	Summer	0.42 +/- 0.01
S.C.		0.80 +/- 0.01
R.H.G.		165 +/- 1

Metric

U-Value	Winter	2.21 +/- 0.01
	Summer	2.35 +/- 0.02
S.C.		0.80 +/- 0.01
R.H.G.		520 +/- 3

Visible and solar data were obtained from the FastScan visible-NIR spectrophotometer and emissivity was calculated from reflectance measurements made on the PE-1420 IR spectrophotometer. The thermal data were calculated using the "Window (3.1)" Program devised by the University of California, Lawrence Berkeley Laboratory.

Thermal data for the IGU are based on the following assumptions:

1. The coating is glazed on the #3 surface.
2. The inside and outside lites are 3 mm thick clear glass.
3. The air gap is 0.5 inches (12.7 mm) and contains air.
4. The window size is 3 feet by 4 feet.
5. The IGU frame is thermally unbroken aluminum (U-value of 10.79)

The data are the average of six samples.

Table III
Super-E III Coating -- July, 1990 Run -- G-6

G-6 Run Data:

Si_3N_4	Power used = 36 kW
	DDR = 700
Base	Line speed for base = 125 IPM x 2 passes
Top	Line speed for top = 90 IPM x 2 passes

Ag	Power used = 3.5 kW
	Line speed = 293 IPM x 1 pass

NiCr	Power used = 0.95 kW
Both Barriers	Line speed = 293 IPM x 1 pass

Estimated Cycle Time for a coater with a load size of 84" x 144" (213 x 366 cm):

- o For HRC-5100 (utilizing three silicon dual C-MAG cathodes each for base and top coat of Si_3N_4).
- o With a Si_3N_4 DDR of 700.

One minute for Si_3N_4 power of 41 kW, a line speed of 175 IPM (inches per minute) or 445 cm for minute.

Table IV
Environmental Testing of Super-E III Coating

Averaged test results of six samples

	<u>Super-E III Coating</u> Average +/- S.D.	<u>Super-E II Coating</u>
Humidity	9.4 +/- 0.4	4.5
Salt Fog	9.3 +/- 0.4	..
UV Exposure		
24 hours	9.5 +/- 0.3	
72 hours	9.2 +/- 0.3	6.8
120 hours	8.8 +/- 0.5	
Ammonium Soak	9.4 +/- 0.3	..
Salt Dot	8.9 +/- 0.2	7.0
Abrasion	9.6 +/- 0.1	0.0

Data for the Super-E II coating are averaged from several test series.

- .. These tests were not run for the initial testing and development of the Super-E II coating.

APPENDIX
Super-E III Coating Environmental Testing

TEST CONDITIONS AND SCORING PROCEDURES

- | | | |
|----|------------------|--|
| A. | Humidity Test | Exposure in a humidity cabinet for 24 hours at 90°C and 98% RH. |
| B. | Salt Fog Test | 20% Salt Fog, 95-98°F for 72 hours. |
| C. | UV Exposure Test | Exposure for 24 hours with cycles of 4 hours UV and 4 hours condensation until failure or 120 hours. |
| D. | Ammonium Test | Samples are placed upright in closed container of 50% ammonium hydroxide solution at room temperature for 5 hours. |
| E. | Salt Dot Test | A 1% salt solution is applied to a filter paper dot placed on the film with the sample placed in a constant humidity environment for 24 hours. |

Evaluation of Tests A to E are based on both microscopic evaluation and emissivity measurements. The details of the evaluations are:

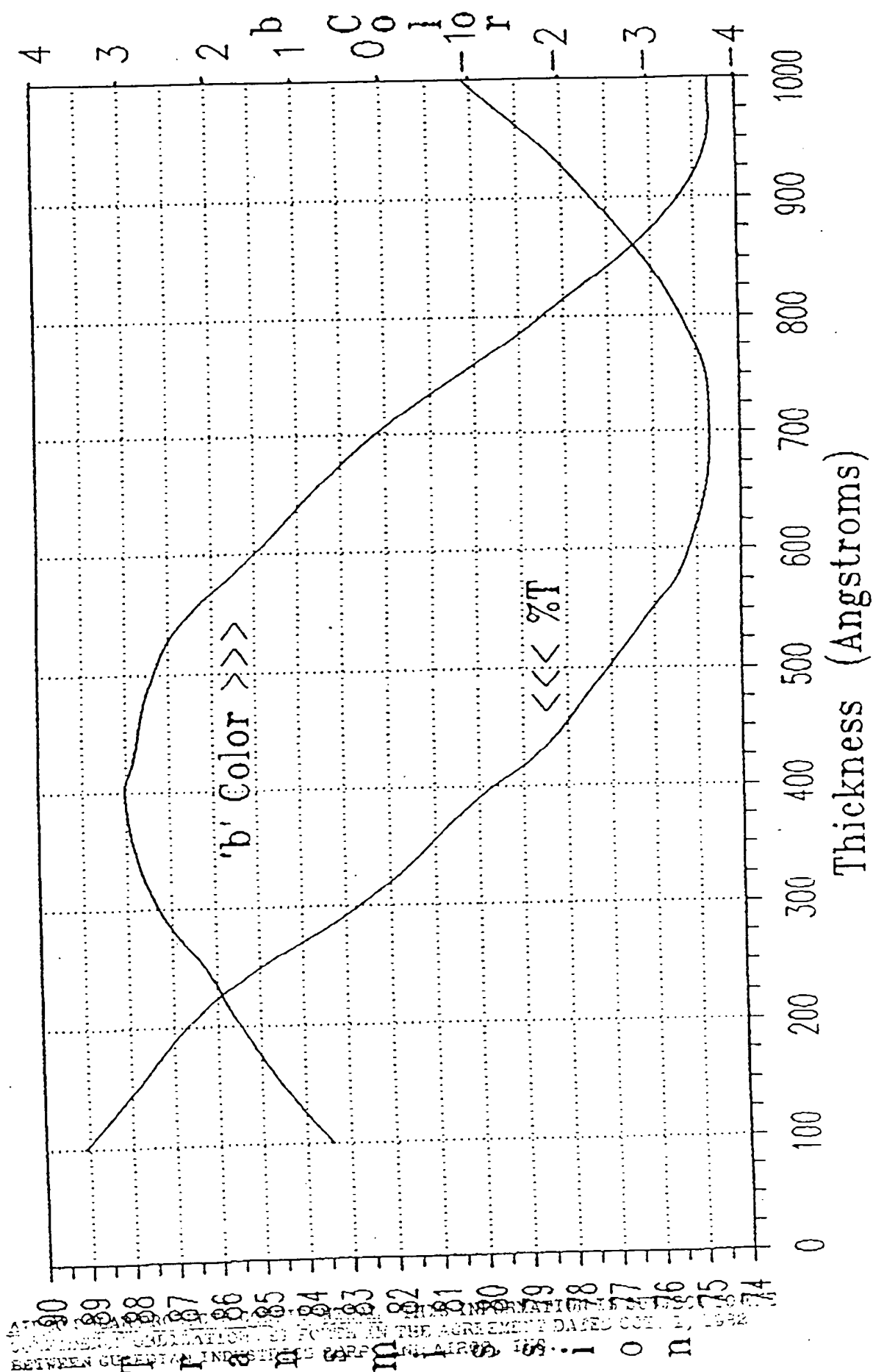
1. Samples are scored for evidence of microscopic corrosion as seen under 200x magnification on a scale of 1 to 10, where 10 is unaffected and 1 is completely corroded.
 2. Measure the change in emissivity due to corrosion. This scoring is based on:
$$\text{Emissivity Score} = 10 (\text{Emiss. Before} / \text{Emiss. After})$$
 3. Recorded scores are an average of 1 and 2.
- F. Taber Abrasion Samples are subjected to a total of 50 revolutions on the Taber abrader, using the standard 500 gram weight and CS-10F wheels.

Evaluation is based on the average number of scratches seen under 50x magnification in 4 inch² areas. Using the equation below gives a score of 0 for more than 55 scratches in a 1" square area and 10 for none:

$$\text{Taber Score} = 10 - \{(\# \text{ Scratches}) \times (0.18)\}$$

Si3N4 on 3mm Glass

G-6 Si CMAG™



AT 1000 ANGSTROMS, THE INFORMATION IS SUBJECT TO THE
 CONFIDENTIALITY OF THE INFORMATION IN THE AGREEMENT DATED OCT. 1, 1992
 BETWEEN GORDIAN INDUSTRIES CORP. AND AIRS, INC.